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(54) Title: WHEY PROTEIN PRODUCT METHOD FOR ITS PRODUCTION AND USE THEREOF IN FOODS

(57) Abstract

Whey protein products having a proportion of their heat denaturable whey proteins denatured using a controlled heating procedure. The products have improved organoleptic characteristics and are useful in the production of a variety of food products especially dairy products such as ice cream.

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WHEY PROTEIN PRODUCT
METHOD FOR ITS PRODUCTION AND USE THEREOF IN FOODS

Technical Field

This invention relates to whey protein concentrate and to food products, such as ice cream utilizing whey protein concentrate as an ingredient. In this application, the term "ice cream" covers full fat ice cream, reduced fat ice cream, low fat ice cream and non-fat ice cream.

Background Art

Whey is a by-product when cheese is produced from milk. After suitable pre-treatment well known to persons skilled in the art, milk is generally treated with a suitable culture to produce curd which is subsequently separated from the remaining liquid, namely dairy whey, and used to make cheese. It is known that whey contains useful proteins, generally known as dairy whey proteins. It is also known that the principal proteins in such whey are ß-lactoglobulin and -lactalbumin. Other proteins include serum derived immunoglobulins. Proteose peptones are also present.

Large quantities of whey are produced as a by-product of cheese production, and various uses for such whey have been developed over the years, mainly as a food ingredient. Such whey usually contains about 12% protein

by weight on a total solids basis. It has become conventional to further treat the whey to provide a product containing at least about 30% protein by weight on a total solids basis, which product is known as whey protein concentrate (WPC). It is usually whey protein concentrate rather than the original fluid whey or dry total whey solids which is used as a food ingredient. A well known process for extracting the proteins from the whey involves heat treating the whey at an acid pH of about 4.5 so as to denature the proteins which then precipitate and are separated from the liquid medium by centrifugation. However, in this process, a significant proportion of the proteins are not denatured and consequently, are lost in the centrifugation step.

Buhler et al, in U.S. 4,265,924 and 4,291,067 disclose a process aimed at improving the protein yield by increasing the amount of protein denaturation and hence precipitatable protein which can then be recovered. The claimed process involves denaturing the proteins present in the whey to an extent of from 35%-70%; removing the non-fat whey constituents from the other contents by ultrafiltration and subjecting the proteins in the retentate to a further heat treatment to effect as complete as possible denaturation of the proteins. The objective is to denature all heat denaturable proteins in the whey and

this is reflected in the severe heat treatment required, such as using a temperature of 95°C-100°C for a period of from 10 to 30 minutes or a temperature of 120°C-160°C for from 5 to 120 seconds. This results in a coagulated product containing particles.

Another problem with such prior process, and this is referred to in Buhler, is that the high temperature used especially in combination with acid pH's have a deleterious effect on the fat present and the organoleptic properties of the resulting protein product. This would preclude its use in many applications.

Whey protein concentrate has been used as an ingredient in ice cream production, namely in the production of full fat ice cream or reduced fat ice cream and has been proposed as an ingredient in low and non-fat ice cream (sometimes called low and non-fat frozen dairy dessert), see for example U.S. Patent 4,840,813 (Greenberg et al.).

However, a major obstacle to such use, particularly in low and non-fat ice cream, has been the fact that the whey protein concentrate tends to cause coagulation of the ice cream mix while it is being pasteurized, with the result that ice cream production has to be shut down to enable the

coagulated material to be removed. This is because, in the past, the whey protein concentrate used has been whey protein concentrate of a conventional kind, namely with at least most, and preferably all, of its protein in the natural state, i.e. undenatured. However, undenatured whey protein can also cause problems, e.g. undesirable gelling during use.

Since persons skilled in the art may interpret the meaning of denaturation and the manner in which denaturation should be measured in different ways, and the value of that characteristic is most important in the present context, percentage denaturation in this application as applied to the present invention means the percentage denaturation when calculated in accordance with the optical based methodology described (unless otherwise stated as "PM" which is defined by reference herein).

Disclosure of Invention

It has now been discovered that the problem of coagulation in ice cream production using whey protein concentrate can be substantially overcome if the denaturation of the whey protein in the whey protein concentrate is controlled during its production so as to be at least about 50% but less than 90% relative to the said

proteins in the raw milk and more preferably from about 60 to about 80%, and more preferably 65 to 75%, when measured by the method described at the end of this specification.

Below about 50%, the prior art problem of coagulation during ice cream production arises. Above about 90%, the ice cream product may have a somewhat sticky, gummy mouth feel which may be unacceptable to some consumers.

In one aspect the present invention provides a process wherein ultrafiltered whey containing substantially undenatured whey protein is subjected to a controlled heating regimen comprising heating at a temperature of less than 90°C for a period of time sufficient to heat denature not less than about 50% but not more than 90% of said heat denaturable protein to produce a whey protein product.

In many instances, non-ultrafiltered whey is readily available and hence in another aspect therefore, the present invention provides a process for preparing a whey protein product comprising:

a) subjecting a whey comprising substantially undenatured whey proteins and lactose to an ultrafiltration step to form a retentate containing whey proteins and a permeate containing a major part of the lactose; and

b) subjecting the retentate to a controlled heating regimen comprising heating at a temperature of less than 90°C for a period of time sufficient to heat denature not less than about 50% but not more than about 90% of said heat denaturable proteins to produce a whey protein product.

In yet another aspect, the present invention provides a process for preparing whey protein concentrate comprising pasteurizing raw milk with resultant denaturation of some whey protein, forming curds in said milk, removing the curds from the remaining whey, subjecting the whey to an ultrafiltration step to remove lactose as permeate, subjecting the ultrafiltered whey retentate to heat treatment to denature further whey protein to cause a total of at least about 50% but not more than 90% of the whey protein to be denatured relative to the raw milk, and concentrating the heat treated whey to produce whey protein concentrate.

Control of the heating regimen or heat treatment, that is the temperature and associated time period, is very important if the product having the desired organoleptic properties is to be achieved. A temperature lower than about 75°C has been found unsuitable bearing in mind that heating periods of greater than say 60 seconds, preferably

30 seconds are to be avoided. Consequently, it is preferred that the ultrafiltered whey be treated at a temperature of from about 75 to 85°C, especially 78 to 82°C for a period of from 5 to 30 seconds. It has been found that a temperature of about 80°C ±0.5 for a time period from 10 to 20 seconds is advantageous. The heat treatment may be effected in any suitable equipment for example, plate or coil heat exchanger or the equivalent. The specific characteristic of the equipment are a factor in determining the optimum temperature/time regimen to be used.

It will be appreciated that the temperatures used to denature the whey proteins according to the present invention are low compared to prior process and hence the regimen is more moderate or gentle resulting in a "tempered" denatured protein product. Hence, in this specification "temperately denatured" means the controlled denaturation of ultrafiltered partially delactosed substantially undenatured proteins at a temperature of not more than 90°C, and preferably, more than 75°C.

Further, the product may be concentrated and hence be a "WPC". It may be used in a liquid or slurry form or dried by usual techniques such as spray drying. The dried product is readily redispersed in water with no loss of the desired characteristics.

It is preferred that not less than 60% but not more than 80%, and especially about 65 to 75%, and most preferably 68 to 72% of the heat denaturable proteins, have been denatured in the process.

The starting whey substrate following the ultrafiltration step and prior to heat treatment preferably has a total solids content of from 5 to 15%, especially 7 to 11% and advantageously 8 to 10%.

In a further aspect the present invention provides a whey protein product comprising temperately denatured whey protein, which is denatured to not less than about 50% and to not more than about 90% based on a total amount of heat-denaturable proteins contained in raw milk.

The protein product of the invention in dry form preferably contains from 30 to 65% whey proteins, partially temperately denatured as detailed herein. Lactose content will generally be in the range of from about 25 to 55%.

The product of the present invention is a partially denatured whey protein useful in a variety of food applications where its fat-substitution and organoleptically pliant and bland characteristics may be used to advantage.

Because of the complex nature of the protein content of whey protein concentrate, the reason for the success of the present invention is not clearly understood. It had previously been believed that the whey protein concentrate used in ice cream production should initially have at least most of its protein in the undenatured state in order to produce acceptable ice cream.

It is possible, although this is just postulation, that the advantages are connected with the relative degree of denaturation of different proteins, such as ß-lactoglobulin and ∝-lactalbumin, when the denaturation is as specified in the present invention. However, the ratio of ß-lactoglobulin to ∝-lactalbumin is not affected by being treated according to the present invention.

It may be advantageous to use raw milk as the basic starting material since the invention may be carried out in a cheese making plant where the whey is produced. However, it may be convenient in other instances to use earlier produced whey provided it is of the desired quality.

Although the type and composition of starting whey substrate may vary, being a natural product and produced by a variety of processes, it is extremely important that it be of a high quality if the desired product is to be

achieved.

The whey should preferably be fresh, substantially uncoloured and preferably has been passed through fine savers. Advantageously, it may be a by-product of the production of brick, cheddar or farmer's cheese but preferably mozzarella.

Preferred characteristics of the whey are as follows:

a) a minimum unadjusted pH of at least 6, not more than 6.5, preferably from 6.25 to 6.35;

b) a titratable acidity of from 0.10 to 0.20%, preferably 0.13 to 0.15%;

c) must not contain a significant amount of non-heat denaturable rennet which has been found to produce off flavours;

d) only heat denaturable enzymes may be present, and especially, no non-heat denaturable lipase may be present, this emanating for example, from parmesan cheese production;

e) hydrogen peroxides, bactericides, antifoaming or de-foaming agents or titanium dioxide should be excluded.

Further, it is most preferred that the whey, upon

being removed from the drain table during cheese production, be cooled to and maintained at 6°C or below, prior to its being processed according to the present invention to form the whey protein product.

In summary, careful control of the starting whey substrate is extremely important to achieving the protein product of the present invention.

It is possible to use a starting whey substrate which is a combination of two or more types of whey for example, a mixture of mozzarella and cheddar whey.

It will be appreciated that a small proportion of the heat denaturable proteins in whey may be, and usually are, denatured during production of the whey from raw milk (herein "raw milk" means untreated milk from which a specific substantially undenatured whey protein is derived). Typically at most 10% or 15% but in extreme cases possibly 20% of the said whey proteins relative to raw milk are denatured. This figure is obtained theoretically since the described optical method of evaluation is not readily applicable in the presence of significant amounts of casein as are present in raw milk. However, because of the importance of controlling the characteristics of the starting whey and the heating

regimen of the whey according to the present invention, it is preferred that such pre-treatment denaturation be kept to a low value for example less than 15% and preferably less than 10% and if possible less than about 5%. For comparison purposes although there is no direct correlation between the two methods, it may be noted that about 15% denaturation measured by the optical method amounts to less than about 5% when measured by alternative precipitation methods ("PM") of evaluation (refer S.J. Rowlands 1938, Determination of Nitrogen Distribution of Milk, J. Dairy Research p.42-26 for the "PM" method.) These denaturation figures may be typical for commercially available WPC's which may be processed according to the present invention.

The whey protein product of the present invention may be used in the production of full fat or reduced fat ice cream or in the production of low or non-fat ice cream and other food products such as yoghurt, sour cream, white sauces, salad dressing, pudding, milk shakes, soft serve ice cream, mayonnaise and other applications where a protein content is required, such as cheese, etc. It is essentially a functional heat "tempered" product in contrast to prior art undenatured or denatured whey protein products. It has improved organoleptic properties as well as a reduced tendency to cause excessive gelling or form lumps or the like when admixed with other food components,

e.g. during the production of ice cream.

In this specification all percentages in formulations, etc. are by weight unless stated otherwise.

Brief Description of Drawings

Preferred embodiments and examples of the invention will now be described, by way of example only, with reference to the accompanying drawings, of which:

Figure 1 is a schematic view of a process for producing cheese and also whey protein concentrate in accordance with the invention,

Figure 2 is a schematic view of a process for making 0% fat ice cream in accordance with the invention,

Figure 3 is a schematic view of a process for making 1% (by weight) fat ice cream in accordance with the invention, and

Figure 4 is a schematic view of a process for making ice cream with 7% (by weight) and higher amounts of fat in accordance with the invention.

Mode For Carrying Out The Invention

Referring to Figure 1, which shows the preparation of whey protein concentrate in accordance with a preferred embodiment of the invention, raw milk at a temperature of from about 3 to about 6°C is preheated in a preheating step 10 to a temperature of from about 43 to about 49°C and then passed to a fat separation step 12 where some fat is separated, the actual amount depending upon the type of cheese to be produced. The preheated fat-reduced milk is then pasteurized in a pasteurization step 14 at a temperature of about 73°C for about 20 seconds, with subsequent cooling to a temperature of from about 32 to about 38°C. The pasteurized fat-reduced milk then passes to a curd forming step 16 where lactic culture is injected and rennet is added in known manner and the contents are cooked and cut to produce curd.

The resultant curd/whey slurry is pumped to curd removal step 18 where raw whey is drained off at a temperature of from about 38 to about 41°C. The curd is subsequently processed into cheese, in this case mozzarella cheese, in any desired manner. At this stage, the protein in the whey is from about 5 to about 10% denatured, relative to the raw milk, most of the denaturation having occurred when the milk was pasteurized in the

pasteurization step 14.

The procedure from hereon was also followed using a mixture of 90% mozzarella and 10% cheddar cheese whey with similar results.

The whey having a pH of about 6.1 solids content of about 6% from curd removal step 18 is pumped to pasteurization step 20 where further pasteurization occurs at a temperature of about 74°C for about 30 seconds, with subsequent cooling to a temperature of from about 50 to about 52°C. This treatment causes further denaturation of the protein such that the protein is then from about 10 to about 15% denatured relative to the raw milk. It will be appreciated that pasteurization steps are carried out for practical handling reasons and to ensure retention of whey quality. In other plant configurations they may not be needed.

The pasteurized whey is pumped to an ultrafiltration step 22 where the whey is ultrafiltered with a membrane having a nominal molecular weight cut-off of 5,000 (such as a KOCHXL-1000™ by KOCH Membrane Systems Inc., Wilmington. MA, U.S.A. The permeate from ultrafiltration step 20 may be used as desired. Most of the lactose in the whey will be in the permeate.

The retentate, namely ultrafiltered whey with a pH of 6.1 and about 9% total solids by weight, is pumped to a heat treatment step 24 where it is subjected to treatment in a plate heat exchanger (made by APV) at a temperature of about 80°C for about 17 seconds. Further and by far the most denaturation occurs during this stage such that the protein in the whey is from about 60 to about 80% denatured, (relative to that in raw milk). This specific time temperature regimen gave a product having a denaturation value of about 71% (which product gave a value in the order of 40% PM. The pasteurized ultrafiltered whey proceeds to a concentration step 26 where evaporation is carried out at a temperature of about 69°C under a vacuum of about 23 inches Hg to concentrate the total solids content to from about 30 to about 32% by weight. After concentration step 26, the whey protein concentrate (WPC) is cooled to about 6°C in a cooling step 28, and may be used in its liquid form. The product was also spray dried for use in its dry form.

The following table details a number of whey protein concentrates of the present invention produced using the procedure generally as described above the products being in liquid form:

	Total Solids %	Acidity %	pH	Protein %	Protein Denaturation % - PM
•	26.03			32.01	37.92
	31.34				40.02
	25.59	0.65	6.10	36.01	52.80
	26.67	0.60	6.21	36.30	39.98
	30.67			36.10	47.98

Lactose in all cases constituted about 50 to 55% of the total solids.

To determine the effects of a more severe heat treatment, the product denoted by "•" was diluted to a total solids content of 15.39 and heated at 120°C for 60 seconds. The resulting protein product was effectively completely denatured giving a protein denaturation value of >70% (PM) and had coagulated and contained readily discernible particles. It was clearly totally unacceptable under the criteria of the present invention.

The denaturation of the whey protein concentrate produced in accordance with the process described above can be controlled so as to be at a value in accordance with the invention by varying the temperature and/or time in the heat treatment step 24 within limits as described above.

The above general procedure was repeated but wherein some specific conditions were varied as given:

EXAMPLE A

Treatment Regimen	78 to 79°C/16 secs
Total Solids	40%
Drier Temperature	74 to 77°C
Protein Denaturation	30 to 40% PM

This gave a dry product.

EXAMPLE B

Treatment Regimen	79 to 80°C/16 secs
Total Solids	31 to 32%
Protein Denaturation	32 to 37% PM

This product was used in its fluid form.

It may be noted that when Example A was repeated but with a temperature regimen of 74°C for 16 seconds and a drier temperature of about 71°C, protein denaturation in the resulting dry product was only 12 to 18% (PM). (The difference in drier temperature was not found to be significant.)

EXAMPLE C

A starting 6% solids whey was evaluated for percentage denaturation prior, and subsequent to, being subjected to the said heat regimen as follows:

	% Denaturation(PM)
Before the Ultrafiltration Step	0.95
Following the Ultrafiltration Step	5.12
* Following Heat Treatment at 80 to 81°C	53.87

* This may be compared with use of a heat treatment at 74°C under otherwise similar conditions which resulted in the whey protein being denatured to a value of only about 11%.

EXAMPLE D

Further studies indicate that small changes in temperature in the heating regimen are important and, for example, have more effect than changes in total solids:

Heating Temperature	Total Solids	Protein Denaturation (PM)
82°C	27.7	49%
82°C	29.2	48%
84°C	24.8	58%
87°C	26.8	61%

Further, the higher temperatures tend to produce products of lesser quality and hence temperatures of about 80°C are preferred. Denaturation values of greater than about 65% (PM) are therefore undesirable.

In a white sauce application, the product of the present invention at say the 2% by weight level based on the total composition, replaces part of the butter or vegetable oil component as well as allowing reduced levels of starch, since the product assists in the creation of a smooth sauce product.

In yoghurt, again, the use of the product of the invention allows a reduction of fat content, and assists, via its gelling properties, in obtaining the desired "body" in the yoghurt.

A sample sour cream utilizing the WPC of the present invention is as follows:

1% Fat Sour Cream

Skim Milk	20,000kg	84.6%
Cream	700	2.9
Dry Blend		
Skim Milk Powder	1,800kg	7.60%
Whey Protein	500	2.10
Starch	450	1.90
Gelatin	71	0.30
Sodium Alginate	60	0.25
Natural Flavour	70	0.22

Processing:

1. Dry blend ingredients.
2. Combine all ingredients and heat to 170 to 175°F (77 to 79°C) for 20 minutes.
3. HTST process at 180 to 185°F (82 to 85°C) for 26 seconds. Homogenize at 1800 psi single stage.

4. Inoculate.

5. Break pH 4.7-4.75.

6. Shear at 15 psi higher than line pressure. Product should smooth out - no graininess should appear.

7. Package and cool.

8. Shelf life ~35 days at 4 to 5°C

As can be seen the denatured whey protein product of the present invention can be used to advantage in many food applications, due in part to its ability to at least partially replace the fat or the like component and to assist in providing body, properties which are demanded by many food items.

Referring now to Figure 2, a process for preparing 0% fat ice cream in accordance with a preferred embodiment of the invention includes blending liquid sweetener, namely high fructose corn syrup, and water in a blending step 30. The resultant blend from blending step 30 is then blended with a first dry blend in a blending step 32, the first dry blend comprising skim milk solids, sweeteners, namely corn syrup solids and dry sugar, and bulking agents, namely tapioca starch and maltodextrin. The resultant blend from blending step 32 is blended with a second dry blend in blending step 34, the second dry blend

comprising stabilizers, namely guar gum, carrageenan, locust bean gum, micro-crystalline cellulose gum, carboxy-methyl cellulose gum and xanthan gum and emulsifiers, namely mono-diglycerides. The resultant blend of dairy ingredients, sweeteners, bulking agents, stabilizers and emulsifiers from blending step 34 is then blended in a blending step 36 with whey protein concentrate from the process described with reference to Figure 1 to form an ice cream mix.

The ice cream mix from blending step 36 is pasteurized in a pasteurization step 38 at about 78°C for about 10 minutes and is then homogenized in a two-stage homogenization step 40. The first stage is carried out at a pressure of about 2500 p.s.i. and the second stage is carried out at a pressure of about 800 p.s.i. The homogenized blend is then cooled in a cooling step 42 to about 4°C, and the cooled blend is then aged for about 24 hours in an aging step 44.

The aged blend is passed to a flavouring step 46 where appropriate flavouring is added, and the flavoured blend is frozen and whipped with an overrun (i.e. increase in volume due to air content) of from about 40 to about 80% in a freezing step 48 to produce 0% fat ice cream which is then extruded from the freezing step 48 at about -6°C. The

ice cream is then hardened in a hardening step 50 until a core temperature (in a two litre container) of about -18°C is reached, this being in about 2 hours.

By way of example, preferred ranges of the ingredients for 0% fat ice cream are as follows:

Ingredients	Per Cent Solids By Weight Of Total Mix
Water	to make up 100%
High Fructose Corn Syrup	7 to 12
First Dry Blend	
Skim Milk Solids	1 to 10
Corn Syrup Solids	2 to 6
Dry Sugar	5 to 8
Tapioca Starch	0 to 2.5
Maltodextrin	0 to 4
Second Dry Blend	
Guar Gum	0.04 to 0.1
Carrageenan	0.01 to 0.04
Locust Bean Gum	0 to 0.05
Micro-crystalline Cellulose Gum	0 to 0.5
Carboxy-methyl-cellulose Gum	0 to 0.4
Xanthan Gum	0 to 0.1
Mono-diglycerides	0 to 0.1

Ingredients	Per Cent Solids By Weight Of Total Mix
From about 30 to about 40% protein by weight on a total solids basis - 60 to 80% Denatured 25 to 40% solids by weight	2 to 7

In a specific example of the invention, the following ingredients for 0% fat ice cream were used:

Ingredients	Per Cent Solids By Weight Of Total Mix
Water	to make up 100%
High Fructose Corn Syrup	10.00
First Dry Blend	
Skim Milk Solids	3.00
Dry Corn Syrup Solids	4.00
Dry Sugar	4.75
Tapioca Starch	2.00
Maltodextrin	3.00
Second Dry Blend	
Guar Gum	0.05
Carrageenan	0.03
Locust Bean Gum	0.04
Micro-crystalline Cellulose Gum	0.3
Carboxy-methyl-cellulose Gum	0.03
Xanthan Gum	0.06
Mono-diglycerides	0.07

Ingredients	Per Cent Solids By Weight Of Total Mix
35% protein by weight on a total solids basis - 71% Denatured 31% solids by weight	7

The liquid blend is blended in a Lanco blender for two minutes at a speed of 1300 r.p.m. The first dry blend is then added slowly and blending is carried out for a further 5 minutes at the same speed. The second dry blend is then added and further blending is carried out for 5 minutes at the same speed. The speed is then reduced to 400 r.p.m., and the whey protein concentrate is added and further blending carried out for 2-3 minutes. The resultant blend is then treated in the manner described above with reference to Figure 2.

Referring now to Figure 3, a process for preparing 1% fat (by weight) ice cream in accordance with a preferred embodiment of the invention includes blending liquid sweeteners, a dairy fat source such as cream and/or butter fat and water in a blending step 52, the liquid sweeteners comprising liquid sugar, liquid corn syrup solids and high fructose corn syrup. The resultant blend from blending step 52 is blended with a dry blend in a

blending step 54, the dry blend comprising skim milk solids and stabilizers, namely guar gum, carrageenan, locust bean gum and micro-crystalline cellulose gum. The resultant blend of dairy ingredients (including fat), sweeteners and stabilizers from blending step 54 is then blended in a blending step 56 with whey protein concentrate from the process described with reference to Figure 1 to form an ice cream mix.

The ice cream mix from blending step 56 is pasteurized in a pasteurization step 58 at a temperature of about 82°C for about 32 seconds and is then homogenized in a two-stage homogenization step 60. The first stage is carried out at a pressure of about 1800 p.s.i. and the second stage is carried out at a pressure of about 700 p.s.i. The homogenized blend is then cooled in a cooling step 62 to about 4°C. The cooled blend is then aged for about 24 hours in an aging step 64.

The aged blend then passes to a flavouring step 66 where appropriate flavour is added and the flavoured blend is frozen and whipped with an overrun of from about 40 to about 80% in a freezing step 68 to produce 1% fat ice cream which is then extruded from freezing step 68 at about -6°C. The 1% fat ice cream is hardened in a hardening step 70 until a core temperature (in a two litre container) of

about -18°C is reached, this being in about 2 hours.

By way of example, preferred ranges of ingredients for 1% (by weight) fat ice cream are as follows:

Ingredients	Per Cent Solids By Weight Of Total Mix
Liquid Blend	
Liquid Sugar	4 to 8
Liquid Corn Syrup Solids	2 to 6
Water	to make up 100%
Cream/Butter Fat	0.5 to 1.5
High Fructose Corn Syrup	7 to 12
Dry Blend	
Skim Milk Solids	1 to 10
Carrageenan	0.01 to 0.04
Guar Gum	0.01 to 0.1
Locust Bean Gum	0 to 0.05
Micro-crystalline Cellulose Gum	0 to 0.5

Whey Protein Concentrate	Per Cent Solids By Weight Of Total Mix
From about 30 to about 40% protein by weight on a total solids basis - 60 to 80% Denatured 25 to 40% solids by weight	2 to 7

In a specific example of the invention, the following ingredients for 1% (by weight) fat ice cream were used:

Ingredients	Per Cent Solids By Weight Of Total Mix
Liquid Blend	
Liquid Sugar	4.75
Liquid Corn Syrup Solids	4.00
Water	to make up 100%
Cream/Butter Fat	0.65
High Fructose Corn Syrup	10.00
Dry Blend	
Skim Milk Solids	4.00
Carrageenan	0.04
Guar Gum	0.016
Locust Bean Gum	0.010
Micro-Crystalline Cellulose Gum	0.008
Whey Protein Concentrate	
35% protein by weight on a total solids basis - 71% Denatured	
31% solids by weight	7.0

The liquid blend is blended in a Lanco blender for about 5 minutes at a speed of about 1300 r.p.m. The

dry blend is then added and further blending carried out for about 5 minutes at the same speed. The speed is then reduced to about 400 r.p.m., the whey protein concentrate is added and further blending carried out for 2-3 minutes. The resultant blend is then processed in the manner described above with reference to Figure 3.

Referring now to Figure 4, a process for preparing ice cream with 7% (by weight) and higher amounts of fat in accordance with a preferred embodiment of the invention includes blending liquid ingredients and water in a blending step 72, the liquid ingredients comprising liquid sugar, liquid corn syrup solids, whey solids, milk solids non fat, and a dairy fat source such as cream and/or butter fat. The resultant blend from blending step 72 is blended with a dry blend of stabilizers and emulsifiers in a blending step 74, the stabilizers being carrageenan, locust bean gum, guar gum and micro-crystalline cellulose gum, and the emulsifiers being polysorbate 80 and mono-diglycerides.

The blend of dairy ingredients, sweeteners, stabilizers and emulsifiers from blending step 74 is then blended in a blending step 75 with whey protein concentrate from the process described with reference to Fig. 1 to form an ice cream mix. The ice cream mix from blending step 76

is pasteurized in a pasteurization step 76 at about 81°C for about 32 seconds and is then homogenized in a two stage homogenization step 78. The first stage is carried out at a pressure of about 1500 p.s.i., and the second stage is carried out at a pressure of about 700 to 800 p.s.i. The homogenized blend is then cooled in a cooling step 80 to about 4°C, and the cooled blend is aged for about 24 hours in a aging step 82.

The aged blend is passed to a flavouring step 84 where appropriate flavouring is added, and the flavoured blend is frozen and whipped with an overrun of from about 30 to about 110% in a freezing step 86 to produce ice cream with 7% fat or higher (for example up to about 20% fat) which is then extruded from freezing step 86 at about -6°C. The ice cream is hardened in a hardening step 88 until a core temperature (in a two litre container) of about -18°C is reached, this being in about 2 hours.

By way of example, preferred ranges of the ingredients for ice cream with 7% (by weight) fat or higher are as follows:

Ingredients	Per Cent Solids By Weight Of Total Mix
Liquid Blend	
Water	to make up 100%
Sucrose Solids	6 to 12
Corn Syrup Solids	3 to 7
Total Fat	7 to 15
Whey Solids	0 to 6
Milk Solids Non Fat	1 to 10
Dry Blend	
Carrageenan	0.01 to 0.04
Locust Bean Gum	0 to 0.05
Guar Gum	0.04 to 0.1
Micro-crystalline Cellulose Gum	0 to 0.4
Polysorbate 80	0 to 0.1
Mono-diglycerides	0 to 0.25
Whey Protein Concentrate (as in the previous examples)	2 to 9

In a specific example of the invention, the following ingredients were used for 7% (by weight) fat ice cream:

Ingredients	Per Cent Solids By Weight Of Total Mix
Liquid Blend	
Water	to make up 100%
Sucrose Solids	10.80
Corn Syrup Solids	7.00
Total Fat	7.20
Whey Solids	3.30
Milk Solids Non Fat	3.875
Dry Blend	
Carrageenan	0.015
Locust Bean Gum	0.0375
Guar Gum	0.06
Micro-crystalline Cellulose Gum	0.03
Polysorbate 80	0.02
Mono-diglycerides	0.08
Whey Protein Concentrate (as in the previous examples)	4.375

In a specific example of the invention, the following ingredients were used for 10% (by weight) fat ice cream:

Ingredients	Per Cent Solids By Weight Of Total Mix
Liquid Blend	
Water	to make up 100%
Sucrose Solids	10.80
Corn Syrup Solids	7.00
Total Fat	10.20
Whey Solids	3.30
Milk Solids Non Fat	3.87

Dry Blend	Per Cent Solids By Weight Of Total Mix
Carrageenan	0.0145
Locust Bean Gum	0.036
Guar Gum	0.058
Micro-crystalline Cellulose Gum	0.029
Polysorbate 80	0.02
Mono-diglycerides	0.08
Whey Protein Concentrate	3.88

(as in the previous examples)

In a specific example of the invention, the following ingredients were used for 15% (by weight) fat ice cream:

Ingredients	Per Cent Solids By Weight Of Total Mix
Liquid Blend	
Water	to make up 100%
Sucrose Solids	12.00
Corn Syrup Solids	4.00
Total Fat	15.00
Milk Solids Non Fat	4.50
Dry Blend	
Carrageenan	0.030
Locust Bean Gum	0.075
Guar Gum	0.120
Micro-crystalline Cellulose Gum	0.060
Polysorbate 80	0.040
Mono-diglycerides	0.160
Whey Protein Concentrate	4.50
(as in the previous examples)	

CALCULATION OF PERCENTAGE DENATURATION

The methodology for calculating the percentage denaturation of the whey protein concentrate will now be described.

In the broadest sense, denaturation of protein

refers to any conformational change in the three dimensional structure of a protein away from its native state. For the purpose of this and in fact most methods which characterize denaturation, the conformational changes must result in a loss of solubility of the protein.

This method involves measuring the protein which remains in solution after a mechanical separation of the precipitated (denatured) portion.

This is a comparative method in which a reference sample is used as a point of "zero denaturation". In most cases, this reference will in fact be partially denatured to a degree which may or may not be known. What is being measured is the percent denaturation in the sample with respect to the reference.

Usually the denaturation of the sample in question is associated with a processing step such as a high heat treatment. In this case, the reference could simply be the sample prior to high heat treatment.

The reference sample is centrifuged to separate out the precipitated proteins. The protein which remains in solution is quantified by UV spectroscopy.

The reference is then completely heat denatured and precipitated proteins are separated by centrifugation. Again, the protein which remains in solution is quantified by UV spectroscopy.

The sample in question is then centrifuged and the protein in solution is measured by UV spectroscopy. By comparing the spectroscopic data for the sample to the data for the undenatured and completely denatured reference, a relative percent denaturation can be calculated.

ULTRAVIOLET SPECTROSCOPY

The amount of UV radiation which a sample absorbs is a function of the concentration of the absorbing components within the sample. This relationship is linear and can be expressed in terms of the Beer-Lambert law.

$$A = bc$$

Where: A = Absorbance

= Extinction Coefficient

b = Path Length

c = Concentration

The extinction coefficient ( ) is a constant for a given substance and the path (b) is a constant for a

given cuvette.

For this method, the absorbances of the aromatic amino acids, tyrosine and tryptophan in the region of 280 nm are used to characterize the concentration of protein in solution. ß-lactoglobulin and ∝-lactalbumin contain these amino acids in different proportions.

Both tyrosine and tryptophan absorb in the 280 nm range. The broad peak which is seen in this region is therefore a composite of absorption peaks of these two amino acids. The two peaks can be viewed separately by looking at the first derivative of the wavelength scan.

Pure solutions of LA and ßLG are used to determine the extinction coefficients of each of these proteins. Accurately prepared mixtures containing different ratios of the two proteins are used to determine composite extinction coefficients for blends.

PERCENT DENATURATION

Once protein concentration in the sample (C_{sample}), the undenatured reference (C_{zero}), and the completely denatured reference ($C_{100\%}$) have been determined, the percent denaturation is determined by the

following equation.

$$\% \text{ Denaturation} = [(C_{zero} - C_{sample})/(C_{zero} - C_{100\%})] \times 100$$

It will be appreciated that the fundamental basis for the degree of denaturation of the denatured protein products of the present invention is the amount of undenatured whey proteins in the milk, from which the whey treated according to the present invention is produced. For convenience, since for example, it may not be possible to readily determine the content of undenatured whey proteins in the said milk, then the optical calculation may be used on the whey to be treated but a correction factor must be applied. If necessary, the above theoretical value may be used.

SAFETY CONSIDERATIONS

This method does not involve any hazardous chemicals. Proper care should be exercised when using the superspeed centrifuge.

APPARATUS

1. Double Beam Scanning UV Spectrophotometer and Quartz Cuvettes (Shimadzu UV160U)
2. Superspeed Centrifuge and Tubes (approx. 25000 G)

3. Boiling Water-Bath

4. 250 ml volumetric flasks

5. Ice-Bath

6. Computer and Spectra-Calc, and RS-1 Software Packages

REAGENTS

1. Distilled Water

2. Purified ∝-lactalbumin (Sigma L-7269)

3. Purified ß-lactoglobulin (Sigma L-0130)

PROCEDURE

1. Determination Of Extinction Coefficient For ∝-lactalbumin

a) Accurately prepare a minimum of 5 solutions (10 ml each) of pure LA ranging from 0.02 to 0.12% (w/w).

b) Set up the parameters of the UV spectrophotometer as follows:

Mode: Wavelength Scan

Wavelengths: 400 to 230 nm

Scanning Speed: Slow

c) Run a baseline correction on the instrument

using distilled water in the reference and sample holders.

d) Scan each solution of LA using distilled water as the reference.

e) Accurately record the peak absorbance in the 280 nm region for each sample. (Use Spectra-Calc to determine peak A) (See "CALCULATIONS" section for determination of)

2. Determination Of Extinction Coefficient For ß-lactoglobulin

a) Accurately prepare a minimum of 5 solutions (10 ml each) of pure ßLG ranging from 0.04 to 0.20% (w/w).

b) Set up the parameters of the UV spectrophotometer as follows:

Mode: Wavelength Scan
Wavelengths: 400 to 230 nm
Scanning Speed: Slow

c) Run a baseline correction on the instrument using distilled water in the reference and sample holders.

d) Scan each solution of βLG using distilled water as the reference.

e) Accurately record the peak absorbance in the 280 nm region for each sample. (Use Spectra-Calc to determine Peak A) (See "CALCULATIONS" section for determination of)

3. Determination Of Composite Extinction Coefficients

a) Accurately prepare 0.1% (w/w) solutions (25 ml of each) of pure LA and βLG.

b) Using these solutions, accurately prepare a minimum of 6 composite samples of varying protein ratios.

c) Set up the parameters of the UV spectrophotometer as follows:

Mode: Wavelength Scan

Wavelengths: 400 to 230 nm

Scanning Speed: Slow

d) Run a baseline correction on the instrument using distilled water in the reference and sample holders.

e) Scan each composite sample with distilled water as the reference. Using Spectra-Calc, determine the maximum peak intensities of the two main first derivative peaks. These peaks will be at approximately 293 and 286 nm. (See "CALCULATIONS" section for the determination of composite)

4a. Analysis Of Reference Sample (Zero Point)

a) Accurately dilute a portion of the reference sample to a solids level of 0.4%.

b) Centrifuge at room temperature for 20 minutes at approximately 25000 G.

c) Set up the parameters of the UV

spectrophotometer as follows:

Mode: Wavelength Scan

Wavelengths: 400 to 230 nm

Scanning Speed: Slow

d) Run a baseline correction on the instrument using distilled water in the reference and sample holders.

e) Scan the supernatant using distilled water in the reference cuvette.

f) Record the peak absorbance (280 nm) as well as the peak intensities of the two main first derivative peaks. (Use Spectra-Calc software)

4b. Analysis Of Reference Sample

(100% Denaturation Point)

a) Fill about 5 250 ml volumetrics with the reference sample.

b) Place the flasks into a boiling water bath and remove 1 every twenty minutes.

For Each Sample:

c) Cool in an ice-bath and use distilled water to bring the volume back to 250 ml.

d) Accurately dilute to 0.4% solids and centrifuge for 20 minutes at approximately 25000 G.

e) Set up the parameters for the UV spectrophotometer as follows:

Mode:	Wavelength Scan
Wavelengths:	400 to 230 nm
Scanning Speed:	Slow

f) Run a baseline correction on the instrument using distilled water in the reference and sample holders.

g) Scan the supernatant using distilled water in the reference cuvette.

h) Record the absorbance at 280 nm and the intensities of the two main first derivative peaks.

i) Continue testing samples until there is no further decrease in the absorbance at 280 nm, i.e. after about 60 minutes.

5. Analysis Of Unknown Sample

a) Accurately dilute the sample to 0.4% solids.

b) Centrifuge for 20 minutes at 25000 G.

c) Set up the parameters of the UV spectrophotometer as follows:

Mode:	Wavelength Scan
Wavelengths:	400 to 230 nm
Scanning Speed:	Slow

d) Run a baseline correction on the instrument using distilled water in the reference and sample holders.

e) Scan the supernatant using distilled water in the reference cuvette.

f) Record the absorbance at 280 nm and the intensity of the two main first derivative peaks.

CALCULATIONS

1. Extinction Coefficient For ∝-lactalbumin

a) Plot a graph of peak absorbance as a function of concentration for the solutions of pure LA.

b) Using RS-1, fit a linear function to the data using the following format:

Absorbance = e x Concentration

c) $_{LA}$ = e

2. Extinction Coefficient For ß-lactoglobulin

a) Plot a graph of peak absorbance as a function of concentration for the solutions for pure ßLG.

b) Using RS-1, fit a linear function to the data using the following format:

Absorbance = e x Concentration

c) $_{ßLG}$ = e

3. Composite Extinction Coefficient

a) Plot a graph of the Ratio LA/βLG as a function of the ratio of the first derivative peaks (A_{293nm}/A_{286nm}) for each of the composite protein samples.

b) Using RS-1, fit a function to the data using the following format:

$$LA/\beta LG = a + [b \times (A_{293nm}/A_{286nm})]^n$$

determine: a, b, and n

4. Concentration Of Reference Sample (Zero Point)

a) Calculate the ratio of the two main first derivative peaks.

$$A_{293nm}/A_{286nm}$$

b) Use this value in the equation derived in step 3 above to determine R, the ratio LA/βLG.

c) Calculate the soluble protein concentration, $^{C}_{(zero)}$, in the undenatured reference sample as follows:

$$^{C}_{(zero)} = A/ \{[1/(R+1)] \times {}_{\beta LG}\} + \{[1-(1/R+1)] \times {}_{LA}\}$$

5. Concentration Of Reference Sample (100%)

For the sample subjected to the longest heat treatment:

a) Calculate the ratio of the two main first derivative peaks.

$$A_{293nm}/A_{286nm}$$

b) Insert this value into the equation derived in step 3 above to determine R, the ratio LA/βLG.

c) Calculate the soluble protein concentration, $c_{(100\%)}$, in the undenatured reference sample as follows:

$$c_{(100\%)} = A/ \{[1/(R+1)] \times \beta LG\} + \{[1-(1/R+1)] \times LA\}$$

6. Concentration Of Unknown Sample

a) Calculate the ratio of the two main first derivative peaks.

$$A_{293nm}/A_{286nm}$$

b) Use this value in the equation derived in step 3 above to determine R, the ratio LA/βLG.

c) Calculate the soluble protein concentration, $c_{(sample)}$, in the undenatured reference sample as follows:

$$c_{(sample)} = A/ \{[1/(R+1)] \times \beta LG\} + \{[1-(1/R+1)] \times LA\}$$

7. Determination Of Degree Of Denaturation

a) Calculate the percent denaturation relative to the reference sample as follows:

$$\% \text{ Denaturation} = [(c_{(zero)} - c_{(sample)}) / (c_{(zero)} - c_{(100\%)})] \times 100$$

As previously mentioned, the percentage denaturation specified in the present invention is the percentage denaturation relative to raw milk from which the undenatured starting whey proteins originate.

Other embodiments and examples of the invention will be readily apparent to a person skilled in the art from the foregoing description of preferred embodiments and examples, the scope of the invention being defined in the following claims.

We claim:

1. A process for preparing whey protein product comprising pasteurizing raw milk with resultant denaturation of some whey protein, forming curds in said milk, removing the curds from the remaining whey, subjecting the whey to an ultrafiltration step to remove lactose as permeate, subjecting the ultrafiltered whey retentate to heat treatment to denature further whey protein to cause a total of at least about 50% but not more than 90% of the whey protein to be denatured relative to that in the raw milk, and concentrating the heat treated whey to produce whey protein product.

2. A process according to claim 1 wherein not more than about 15% of the whey protein relative to that in the raw milk has been denatured when the whey has been pasteurized prior to being ultrafiltered.

3. A process according to claim 1 wherein the ultrafiltered whey retentate is heat treated to denature further whey protein to cause a total of from about 60 to about 80% of the whey protein relative to that in the raw milk.

4. A process according to claim 1, 2 or 3 wherein

the ultrafiltered whey is heated at a temperature of at most 95°C for a period of from 5 to 60 seconds.

5. A process according to claim 1, 2 or 3 wherein the ultrafiltered whey is heated at a temperature of from 75°C to 90°C for a period of from 5 to 30 seconds.

6. Whey protein product prepared by the process of claim 1, 2 or 3.

7. A process for preparing a whey protein product comprising subjecting ultrafiltered whey containing substantially undenatured whey protein to a controlled heating regimen comprising heating at a temperature of less than 90°C for a period of time sufficient to effect heat denaturation of not less than about 50% but not more than about 90% of said heat denaturable protein in raw milk, to produce a whey protein product.

8. A process according to claim 7 wherein the heating regimen comprises heating at a temperature of from about 75°C to about 90°C for not more than 30 seconds to obtain a protein product wherein from about 60% to about 80% of said denaturable whey protein is denatured.

9. A process according to claim 8 wherein the about

70% of said denaturable whey protein is denatured.

10. A process for preparing a whey protein product comprising:

a) subjecting whey containing substantially undenatured whey proteins and lactose to an ultrafiltration step to form a retentate containing whey proteins and a permeate containing part of the lactose; and

b) subjecting the retentate to a controlled heating regimen comprising heating at a temperature of not more than 90°C for a period of time sufficient to heat denature a total of not less than about 50% but not more than about 90% of said heat denaturable proteins in raw milk to form a whey protein product.

11. A process according to claim 7 or 10 wherein the temperature is at least 75°C.

12. A process according to claim 7 or 10 wherein the temperature is from 75°C to 85°C.

13. A process according to claim 10 wherein the temperature is from 78°C to 82°C.

14. A process according to claim 13 wherein the temperature is 80°C ± 0.5.

15. A process according to claim 7 or 10 where said heating is effected for up to 60 seconds.

16. A process according to claim 14 wherein said heating is effected for from 5 to 30 seconds.

17. A process according to claim 16 wherein said heating is effected for from 10 to 20 seconds.

18. A process according to claim 7 or 10 wherein the temperature regimen is such that the said whey protein is from 60% to 80% denatured.

19. A process according to claim 18 wherein the temperature regimen is such that the said whey protein is from 65% to 75% denatured.

20. A process according to claim 19 wherein the temperature regimen is such that the said whey protein is from about 70% to 72% denatured.

21. A process according to claim 7 or 10 wherein the starting whey has a minimum pH of from 6 to 6.5.

22. A process according to claim 21 wherein the starting whey has a pH of from 6.25 to 6.35.

23. A process according to claim 22 wherein the starting whey has a pH of about 6.1.

24. A process according to claim 7 or 10 wherein the starting whey has a titratable acidity of from 0.10 to 0.20%.

25. A process according to claim 24 wherein the starting whey has a titratable acidity of from 0.13% to 0.15%.

26. A process according to claim 10 wherein the whey, following its production in a cheese making process is cooled and maintained at a temperature of less than about 6°C prior to its being used in the process.

27. A process according to claim 10 wherein the why is a by-product from the production of mozzarella cheese.

28. A process according to claim 10 wherein the whey is a mixture of wheys produced as a by-product in the production of mozzarella and cheddar cheese.

29. A process according to claim 7 or 10 wherein the heat denaturable protein in the starting whey is less than 15% denatured.

30. A process according to claim 28 wherein the heat denaturable protein in the starting whey is less than 10% denatured.

31. A process for preparing a whey protein concentrate comprising:

a) subjecting the whey containing whey proteins, at most 15% of which are denatured, and lactose to an ultrafiltration step to form a retentate containing whey proteins and a permeate containing part of the lactose;

b) subjecting the retentate to a controlled heating at a temperature of from 75°C to 85°C for a period of from 5 to 30 seconds to ensure that from about 65% to 75% of the total heat denaturable whey proteins are denatured.

32. A process according to claim 31 wherein the heating regimen comprise heating at a temperature is from about 78°C to 82°C for a from about 10 to 20 seconds.

33. A process according to claim 31 or 32 wherein the said whey proteins are from about 68% to 72% denatured.

34. A process according to claim 32 wherein the heating regimen comprises heating at a temperature of about 80°C ±0.5°C for a period of from 15 to 18 seconds and the

said whey proteins are about 70% to 72% denatured.

35. A whey protein concentrate containing from about 30% to 40% by weight total solids, from about 30% to 40% by weight of said solids being whey proteins and at least from about 50%, but not more than 90%, of the heat denaturable whey proteins being denatured relative to that in raw milk.

36. A product according to claim 35 wherein from about 60% to 80% of the whey protein is so denatured.

37. A product according to claim 36 wherein about 70% of the whey protein is denatured.

38. A heat denatured whey protein product comprising temperately denatured whey protein which is denatured to not less than about 50% to not more than about 90% based on a total amount of heat-denaturable proteins contained in raw milk.

39. A product according to claim 39 wherein from about 65% to 75% of the said whey proteins are denatured.

40. A product according to claim 39 wherein the said whey proteins are from 68% to 72% denatured.

41. A product according to claim 39 wherein said whey proteins are about 71% denatured.

42. A product according to claims 39, 40 or 41 which comprises:

a) from 30 to 65% of said temperately denatured whey protein; and

b) from 25 to 55% of lactose.

43. A product according to claim 39, 40, or 41 which comprises from 30 to 40% of said temperately denatured whey protein and from 45 to 55% lactose.

44. A process for preparing ice cream including forming an ice cream mix as an aqueous blend of solids comprising whey protein concentrate whose solids are in an amount from about 2 to about 9% by weight of the solids in the blend, said whey protein concentrate solids containing from about 30 to about 40% by weight whey protein and having at least about 50% but not more than 90% of its whey protein content denatured relative to raw milk, and processing said mix to form ice cream.

45. A process according to claim 44 wherein the whey protein concentrate has from about 60 to about 80% of its whey protein content denatured.

46. A process according to claim 44 wherein said aqueous blend is prepared by first blending ingredients other than said whey protein concentrate, and then blending in said whey protein concentrate.

47. Ice cream prepared by a process according to any one of claims 44-46.

48. Ice cream prepared by a process according to any one of claims 44-46 and containing less than about 5% by weight fat.

49. Ice cream prepared by a process according to any one of claims 44-46 and containing about 1% by weight fat.

50. A food product having a fat or like content wherein at least part of said fat content is replaced by a product comprising temperately denatured whey protein which is denatured to not less than about 50% to not more than about 90% based on a total amount of heat-denaturable proteins contained in raw milk.

51. A food product according to claim 50 which is an ice cream.

52. A food product according to claim 50 which is a sour cream.

53. A food product according to claim 50 which is a white sauce.

54. A food product according to claim 50 which is yoghurt.

FIG. 1

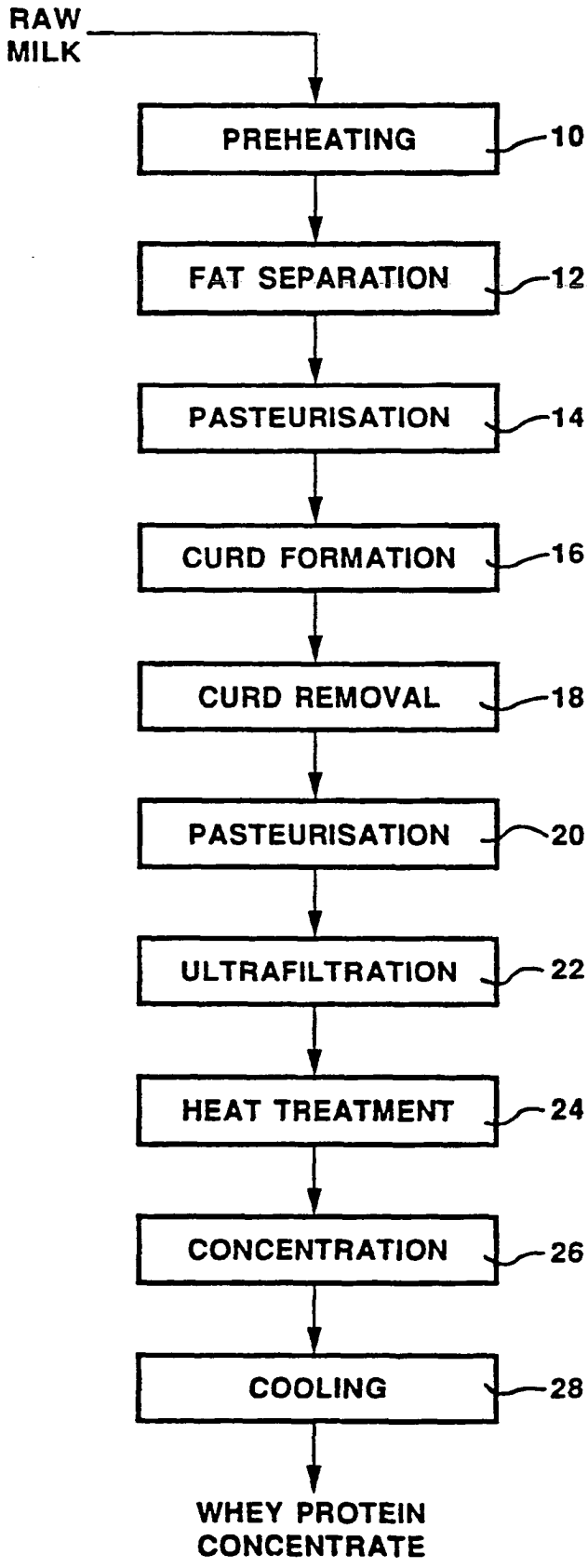


FIG. 2

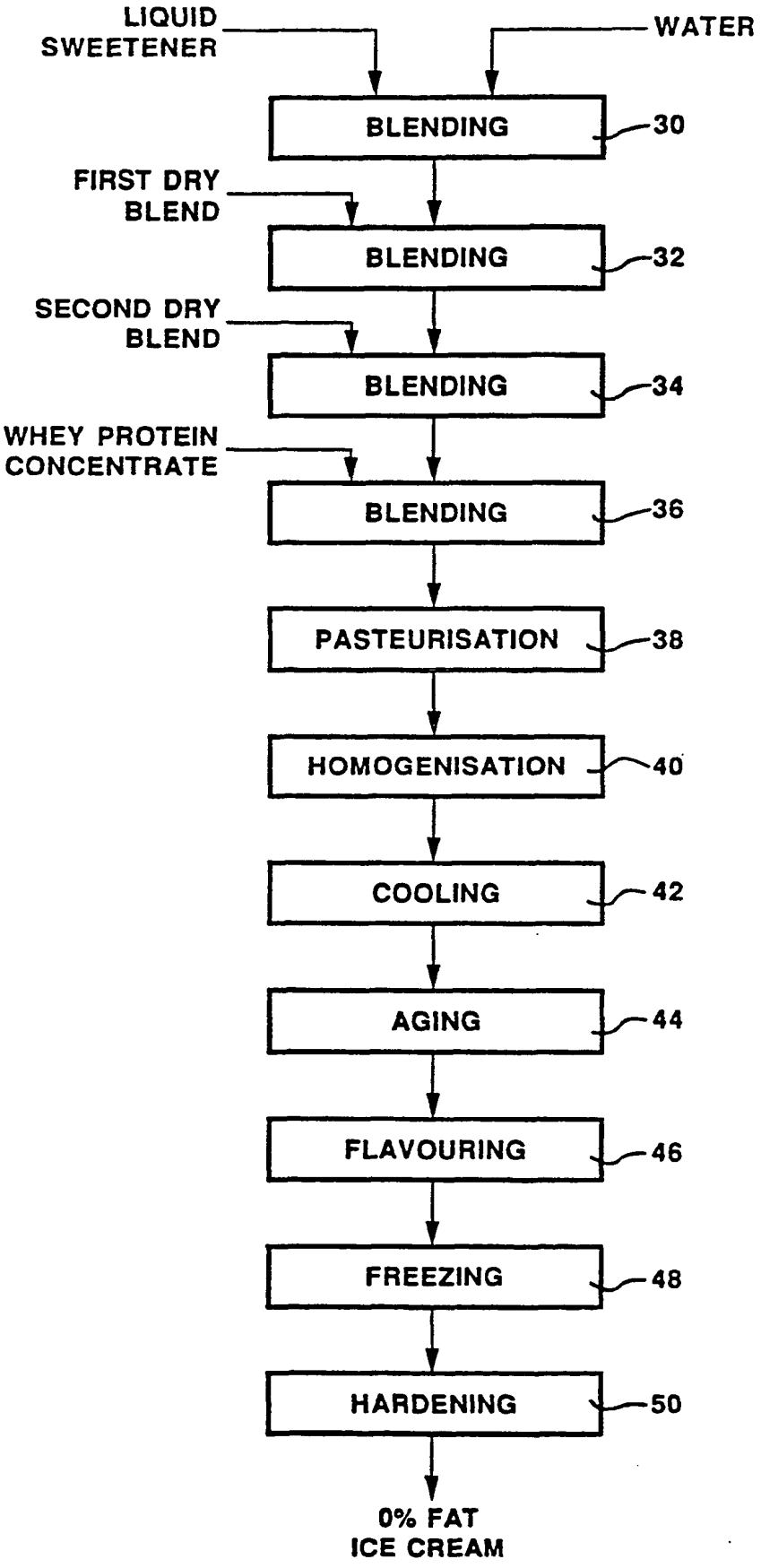


FIG. 3

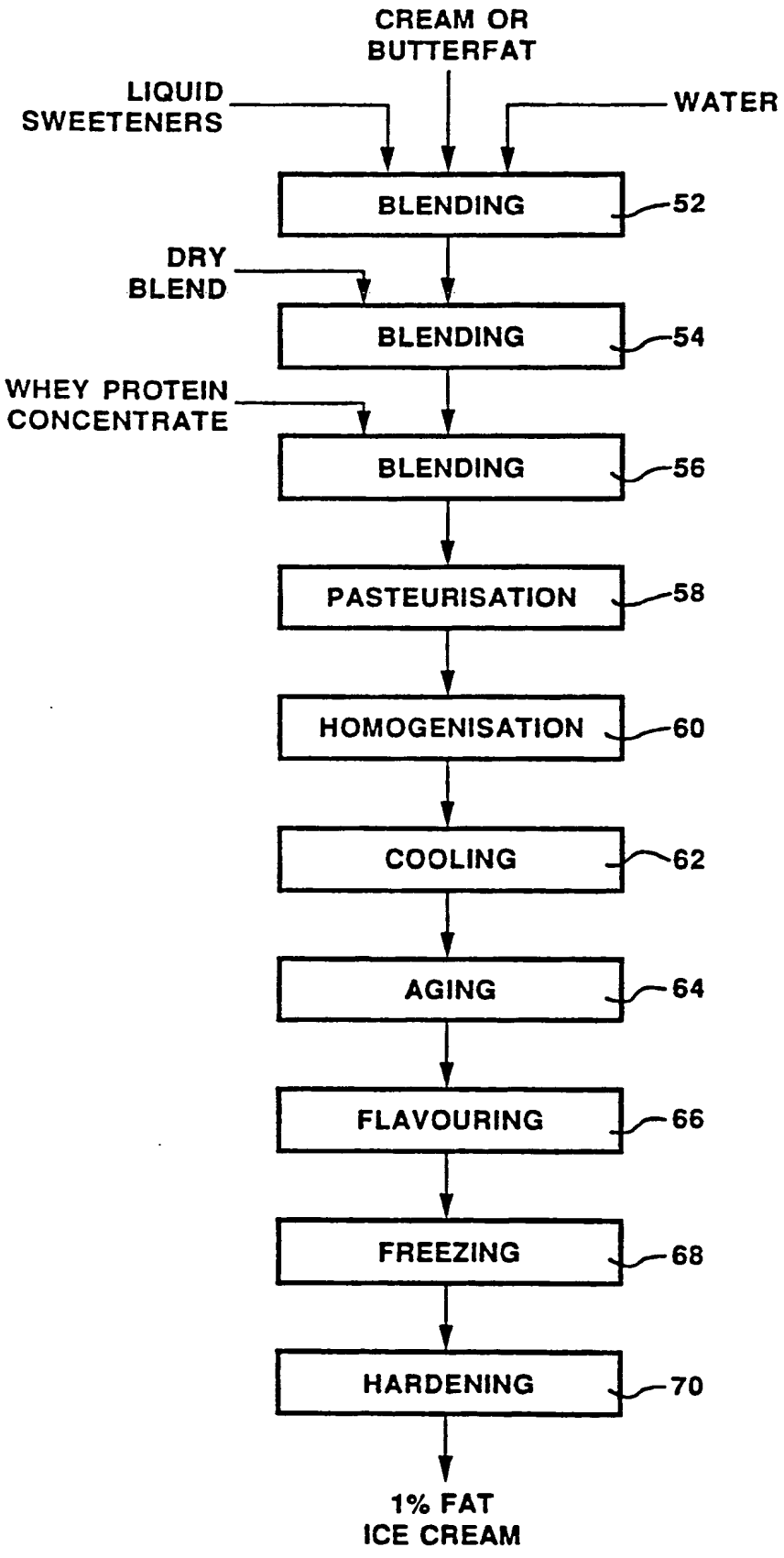
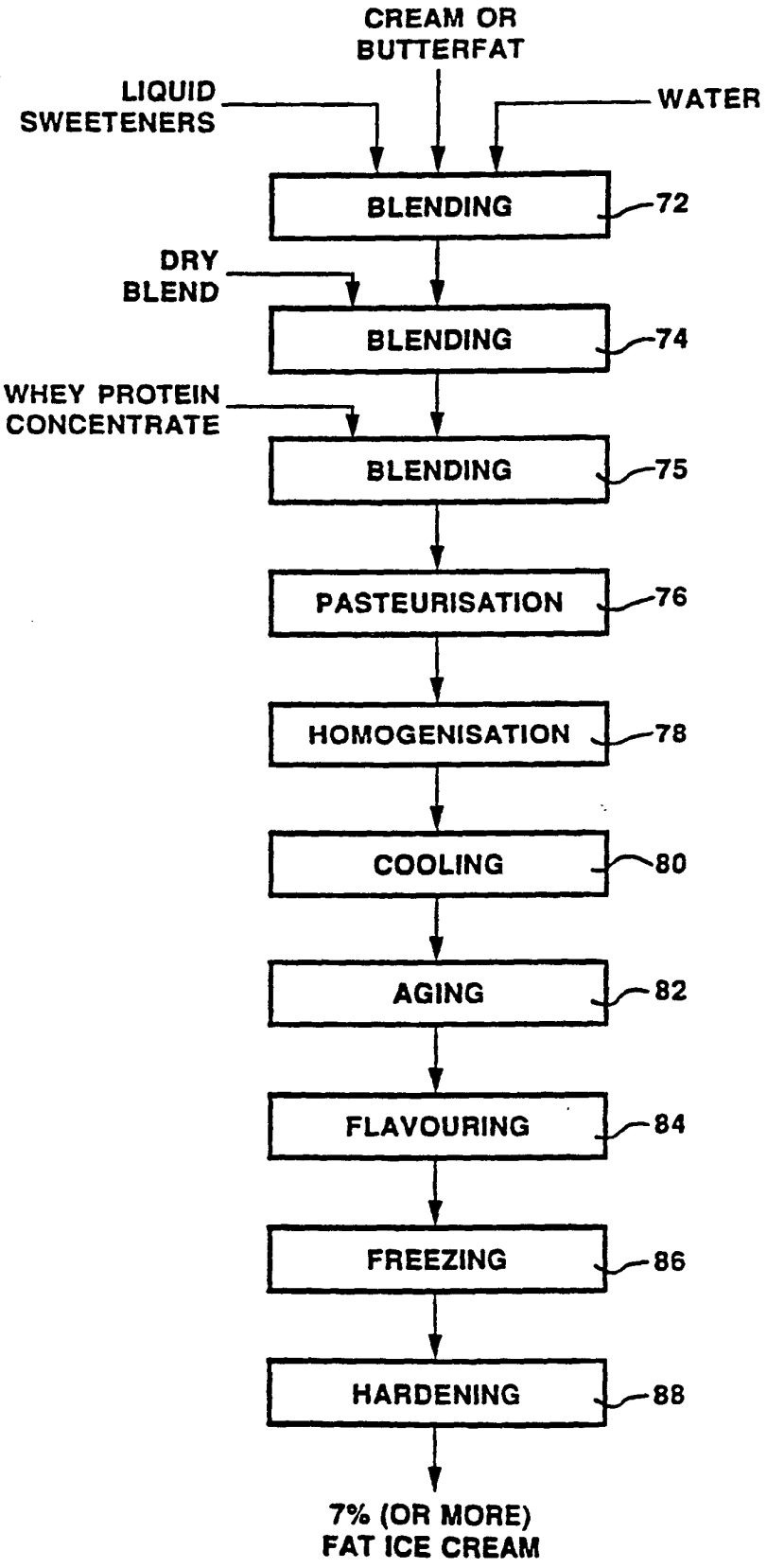


FIG. 4


CREAM OR BUTTERFAT
LIQUID SWEETENERS
WATER
BLENDING
72
DRY BLEND
BLENDING
74
WHEY PROTEIN CONCENTRATE
BLENDING
75
PASTEURISATION
76
HOMOGENISATION
78
COOLING
80
AGING
82
FLAVOURING
84
FREEZING
86
HARDENING
88
7% (OR MORE) FAT ICE CREAM

INTERNATIONAL SEARCH REPORT

International Application No PCT/CA 92/00210

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl. 5 A23J3/08; A23J1/20; A23G9/02; A23L1/305
A23L1/39; A23C9/13; A23C13/16

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl. 5	A23J ; A23G ; A23C

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category °	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
X	EP,A,0 029 370 (STAUFFER CHEMICAL CO.) 27 May 1981	1,3, 4-20,29, 30-42
Y	see claims 1,4,6,15 see page 7, line 4 - page 8, line 11 see page 11, line 14 - line 27 see page 15, line 30 - page 16, line 22 see page 17, line 23 - page 18, line 1 see page 18, line 30 - line 8 see page 23, line 2 - line 9 see example 2; table IV --- -/--	21-23, 32,44, 47-54

° Special categories of cited documents :[10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establisb the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
23 SEPTEMBER 1992	06. 10. 92

International Searching Authority	Signature of Authorized Officer
EUROPEAN PATENT OFFICE	VUILLAMY V.M.L.

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